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Ultra-Wide Bore 900 MHz Bucket Test, Installation, and Commissioning

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The world's first 105 mm bore, 900 MHz NMR system will soon begin its commissioning phase at the NHMFL in Tallahassee. The entire NMR magnet system has been designed, built, and tested. The cryostat is nearly completed. Facilities have been upgraded. NMR probes and equipment are being prepared and we are on the threshold of commissioning. Since our last report in the spring of 2002 (*NHMFL Reports*, **9**(2), 2002), at which time the magnet system had just undergone 4.2 K testing, much has been accomplished. Specifically, the final 2.3 K bucket testing has been completed. Fabrication of the 1.8 K superfluid helium cryostat is nearly finished. The 900 MHz magnet and cryostat assembly has been relocated to the final user area, and preparations are being made for commissioning. This report describes the progress made over the past year and a half of the NHMFL Wide Bore 900 MHz NMR System and facilities and goes into some detail on the designs of the cryostat and NMR measurement systems.

Completion of the Bucket Test

At the end of August 2002, the bucket test of the magnet system was completed. The magnet system was tested in a temporary "bucket" cryostat to confirm operation of each of the sub-systems assembled together as an integrated unit for the first time. The bucket test confirmed operation of the magnet system, the quench protection system, and all the associated superconducting switches, coils, shims, and electronics. The bucket cryostat provided saturated 2.3 K helium and relatively easy access to the test assembly to allow modifications required as determined by the system testing. The bucket cryostat, due to its easy access requirement, could not provide the sub-cooled, 1.8 K helium environment of the permanent cryostat, and it was realized that this cryogenic limitation might prevent achievement of 900 MHz. ***The system, however, was charged to 875 MHz on its first attempt at 900 MHz!*** Subsequent attempts reached 810 MHz and 843 MHz and showed no indication of magnet system degradation as a result of quenching. Several modifications were made to the quench protection system and the associated subsystems during the bucket testing. The superconducting shim sets were fully characterized and their shim capabilities were confirmed. The decay rate of the magnet system was measured and found to be higher than specified. As a result, a current injection method of correcting for field drift was incorporated into the final system design and will be tested during the upcoming commissioning phase. Overall, the bucket test was very successful. It characterized the basic capabilities of this first-of-its-kind system and allowed modifications to the integrated magnet and quench detection system before installation into the permanent cryostat. But most promising, it nearly achieved its full operational field in the relatively low thermal stability provided by the saturated 2.3 K helium environment.

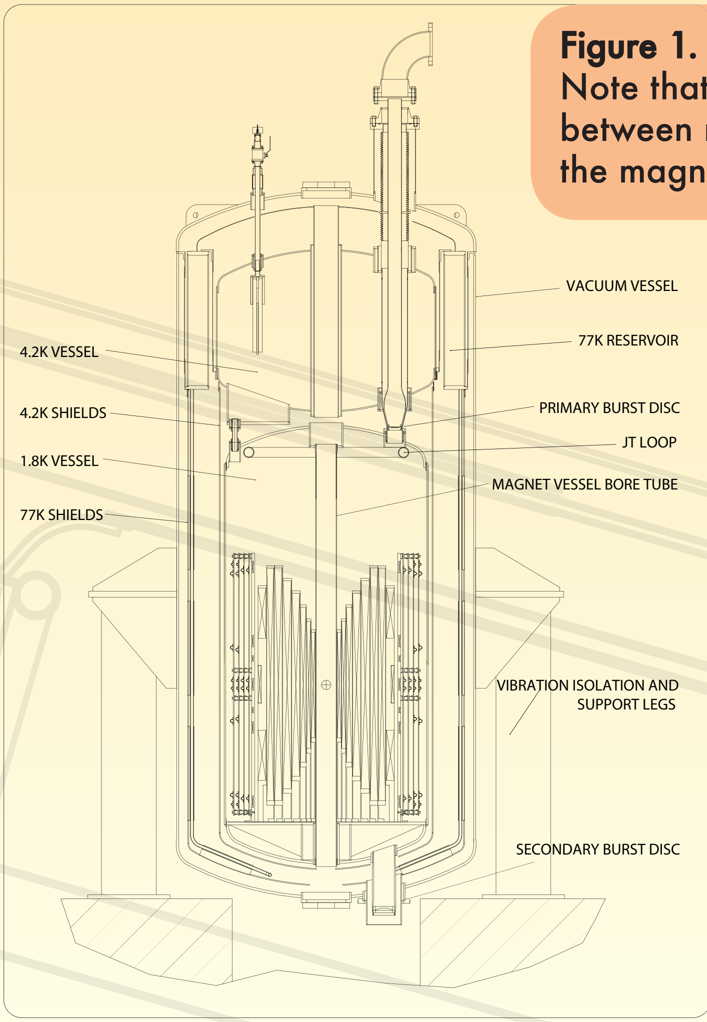

Figure 1. Some of the key features of the cryostat. Note that only two of the eight service penetrations between room temperature, the 4.2 K reservoir, and the magnet vessel are shown.

The Permanent 900 MHz NMR Cryostat

The permanent 900 MHz NMR cryostat, designed by Steve Van Sciver, Kurt Cantrell, and Scott Welton, is described as follows. The 900 MHz NMR magnet cryostat consists of a vacuum vessel and two liquid helium reservoirs as shown in the assembly schematic in Figure 1. The upper reservoir, which is made of 316 stainless steel, contains a volume of saturated liquid helium at 4.2 K. There are eight penetrations from room temperature to the 4.2 K reservoir, one each for: (1) the main current leads, (2) quench and switch heaters, (3) the auxiliary power leads, (4) the burst disc assembly, (5) the cool down port, (6) the instrumentation probe, (7) voltage taps, and (8) the JT refrigerator. The 4.2 K reservoir consists of approximately 1,100 liters of saturated helium (He I) and is sized so that the hold time between refills is 23 days.

The lower reservoir, also made of 316 stainless steel, holds the magnet in a volume of sub-cooled superfluid helium (He II) at 1.8 K and one atmosphere. The magnet reservoir has an enclosed volume of approximately 3,500 liters, which contains the 900 MHz magnet system assembly and approximately 2,400 liters of He II. The 1.8 K operating temperature is maintained by a JT refrigeration system. This component consists of a copper heat exchanger containing saturated 1.8 K He II, which is in thermal contact with the sub-cooled helium in the magnet vessel. The temperature of the saturated He II in the heat exchanger is controlled by regulating the vapor pressure above the bath with a vacuum pump located at room temperature. The liquid level in the heat exchanger is controlled by a feedback loop that monitors the liquid level and controls the JT valve position.

Thermal radiation shielding is provided at two temperatures, 4.2 K and 77 K. The magnet reservoir is surrounded on its bottom and outer surfaces by the 4.2 K shield. The 4.2 K shield is conductively cooled and thermally anchored to the 4.2 K reservoir. Due to a lack of available space, there is no 4.2 K radiation shield on the inner bore of the magnet vessel or on the secondary burst disc nozzle. The 4.2 K reservoir and shield are in turn surrounded on their outer and inner surfaces by 77 K shields, which are actively cooled by liquid nitrogen (LN_2) beneath the 77 K reservoir and conductively cooled above the 77 K reservoir and in the 4.2 K vessel bore. The 77 K shield below the reservoir is a continuous cylinder of ¼" thick aluminum. A custom designed and fabricated composite 77 K shield is located on the inner diameter between the magnet vessel and warm bore tube and is actively cooled by LN_2. The 560 liter toroidal 77 K reservoir is sized for a 24-day hold time.

The weight of the magnet and cryostat assembly is supported internally by composite support cylinders and stainless steel straps and externally by four support columns, which also provide vibration isolation. Within the cryostat, two G10 composite support cylinders and six stainless steel straps carry the weight of the magnet assembly from the top. One composite cylinder and six stainless steel straps are located between room temperature and 77 K and carry a load of approximately 23,000 lbs. A second composite cylinder is located between 77 K and 4.2 K and carries a load of approximately 17,000 lbs. The cryostat is supported externally by four Fabreeka® support columns and vibration isolation units. The vibrational stability requirement is met by the vibration isolation pads located on the support columns and by six horizontal anti-vibration straps located between the vacuum vessel and the magnet vessel near the bottom of the magnet vessel.

The cryostat is built around the magnet on site at the NHMFL. Subassemblies were fabricated at the cryostat supplier then shipped to the NHMFL for final assembly in Cell 16 which has a 25 T overhead crane available. After the magnet assembly was attached to the 4.2 K vessel and the 1.8 K vessel was welded in place, it was transported to its final operation area in NM112 for final assembly of the thermal shields and vacuum vessel.

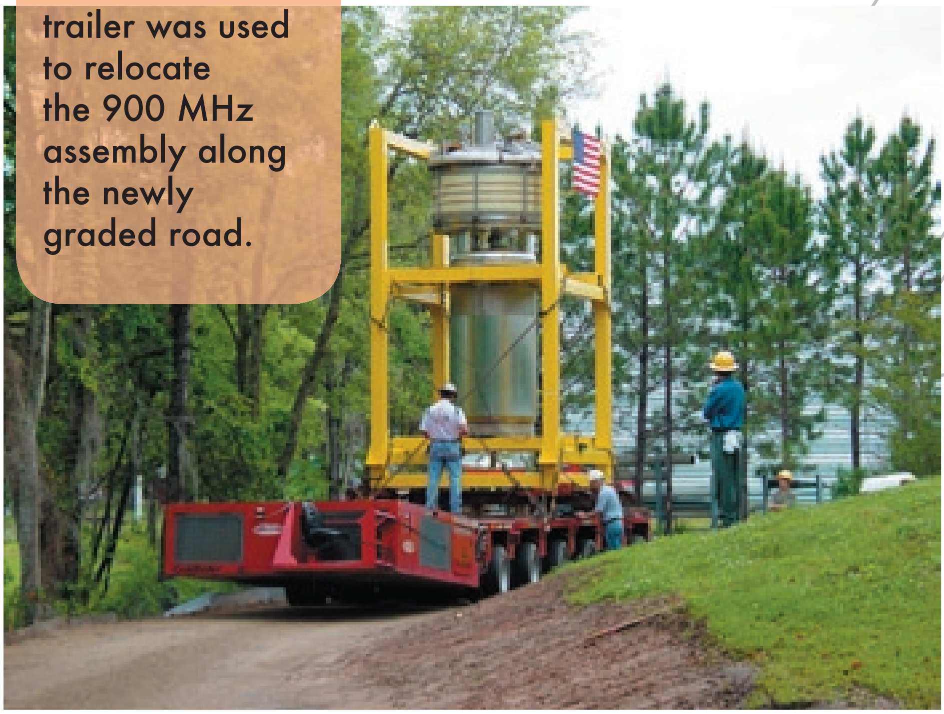

Figure 2. A Goldhofer trailer was used to relocate the 900 MHz assembly along the newly graded road.

Fabrication of the Permanent Cryostat and Relocation into the Final User Area

Since bucket testing, the primary emphasis has been on fabricating the permanent cryostat around the magnet assembly. These activities at the NHMFL, led by Iain Dixon, have involved many hundreds of tasks, but the basic steps completed before relocation to the final user area were as follows. The 4.2 K vessel assembly, received as a fabricated unit from the cryostat supplier, comprises essentially the upper half of the cryostat and weighs approximately 6,000 lbs. This assembly was lowered over the magnet and welded to the magnet vessel bore tube. Welding to the bore tube was quite a challenging exercise considering the massive, large diameter 4.2 K vessel assembly had to be located and balanced in mid-air above the magnet assembly to within a few ten thousandths of an inch to mate to the partially inserted bore tube. Note that the magnet vessel bore tube is designed inside the magnet assembly with extremely tight tolerances to provide maximum space to the NMR scientists. After the bore tube weld was leak checked—while hanging in midair—the entire 6,000 lb assembly was lowered into its final position by sliding the bore tube inside the innermost superconducting coil with nearly zero clearance between the two. Finally, the outer shell of the magnet vessel was welded in place and leak checked at which point the magnet and cryostat assembly was ready for relocation to the final user area.

In early April 2003, the magnet and cryostat assembly was safely relocated to the opposite end of the laboratory. Up until this point, the cryostat assembly required the use of the high bay area and crane in the Cell 16 fabrication area. Attachment of the next hardware items, the thermal shields, however, would have raised the risk of damaging the 16 foot tall, 23,000 lb. assembly during the relocation process. After exploring many options for the method and the path by which to move the assembly, it was finally decided to subcontract the relocation

Figure 3. The 4.2 K Shields were hung around the magnet vessel after the entire assembly was moved to the final user area.

task to Barnhart Crane and Rigging. Barnhart used a Goldhofer trailer to relocate the magnet and cryostat assembly, and a road was newly graded behind the lab to clear the way for the move. The Goldhofer trailer can independently raise and lower multiple sections of the trailer to maintain the transported load as level as possible. A custom transportation frame was designed and built at the NHMFL specifically for the move and for the remaining fabrication tasks required in the final user area. A practice run with a full size test load was performed the day before the actual relocation was performed, and special rails were laid down to move the assembly out of and into the buildings at each end. Vibration measurements during the move indicated the typical vibration was consistently less than 0.02 g and only during one event did it reach just over 0.06 g. In all cases, the recorded vibrations were less than the specified maximum allowable of 0.1 g. Figure 2 shows a picture of the 900 MHz assembly being moved by the Goldhofer in its transportation frame along the newly graded road. More details of the relocation process can be seen in photographs shown in the last issue of *NHMFL Reports* (**10**(2), 2003). The relocation process resulted in a successfully and safely transported 900 MHz assembly.

Presently, the final fabrication of the 900 MHz cryostat is being completed in NM112. The 77 K traced bore tube has been installed. The 4.2 K shields and 4.2 K multi-layer insulation (MLI) have been attached as shown in Figures 3, 4, and 5. The 77 K shields and 77 K MLI are the present focus of activities. This will be followed immediately by attachment of the vacuum vessel and vibration isolation and support legs. Finally, the yellow relocation frame will be removed, the user platform will be installed, the utilities and controls will be connected, and the commissioning phase will begin.

Facilities Activities

It is important to note the key role that the NHMFL Facilities group, led by John Kynoch, is playing in the installation of the 900 MHz system with respect to utilities, control, and safety. The Facilities group has literally laid the groundwork to allow the system to be relocated and installed. First of all, the path chosen to relocate the magnet was an uneven, rocky road until Facilities led the activities to grade the path to ensure a safe and secure relocation process for the magnet as well as provide an environmentally friendly road construction process. Furthermore, the Facilities group has been working closely with the 900 MHz team on a daily basis to develop the utilities required of an NMR user facility including the programming and installation of the control logic that will help run the magnet system and ensure the safety of equipment and personnel. Facilities has also assisted in the design and installation of the helium recovery and vacuum systems. But perhaps most challenging, ***Facilities has upgraded the HVAC to attempt to meet a temperature stability requirement of +/- 1 degree Fahrenheit throughout the 900 MHz user area, which will maximize the ability to perform the best NMR science.***

Figure 4. Multi-layer insulation (MLI) required between the 4.2 K and 77 K shields was fabricated into five-layer blankets in NM112 before being applied to the assembly. Shown here paying careful attention to an accurate and clean process are Lee Marks, Steve Lydzinski, and Don Richardson from left to right.

Figure 5. The MLI has to be custom cut to match the dished head contours and to interface with the secondary burst disk and bore tube penetrations at the bottom of the shields.

The Commissioning Phase

The installed wide bore 900 MHz system will undergo extensive commissioning. During the commissioning, we will fully characterize and fine tune the operation of the magnet, shim sets, bucking coils, cryogenics, data acquisition, active quench protection system, and NMR operations in their final location. The first item to be confirmed during the commissioning phase is the safety of the pressure vessel. This will be performed by a proof pressure test at 77 K to 6 atmospheres, 20% over the design pressure. After that, the system will be warmed up to allow installation of a 5 atmosphere secondary burst disc as a redundant pressure vessel safety precaution. Next, the system will be cooled again, filled with liquid 4.2 K helium, and then cooled further to 1.8 K. At this point, the magnet will be charged to 900 MHz. It is typical for a superconducting NMR magnet to undergo a training period of quenches until full field is reached, and it is likely that the 900 MHz system will also experience some training quenches.

Once the magnet is at field, a range of activities will be carried out to prepare for NMR application science as follows. The magnetic field will be homogenized in several steps to achieve the high uniformity required for many NMR studies. First, the current in the superconductive shim coils will be adjusted based on iterative NMR mapping of the field. Then a custom-designed room temperature shim coil insert will be installed and tested. The room temperature shim coils will leave a full 89 mm inner diameter for user instrumentation. The novel current injection system will be tested and adjusted to compensate for field drift. Design calculations indicate that the current injection will produce a minor loss of field homogeneity that may be significant for some solutions experiments. The shim and current injection power supply is being designed to allow for continuous compensation of this effect, once it has been measured. When these basic mapping and adjustment operations are complete, installation and testing of the NMR console and sample probes can proceed.

The NMR science activities, led by Tim Cross and Bill Brey, are described as follows for the commissioning phase. A new, multichannel, high-power NMR console and suite of sample probes has already been partially delivered by Bruker Instruments. It includes a triple axis gradient HCN probe for solution NMR of proteins, a triple resonance CPMAS probe for materials chemistry and solid state NMR, and a set of imaging probes for biomedical applications. Specialized probes for membrane protein analysis are under development at NHMFL through an In-House Research Program-sponsored collaboration with leading NMR scientists. Extensive testing and adjustment of the equipment and demonstration of NMR techniques by both NHMFL staff and Bruker engineers will be carried out during the commissioning phase. The NMR experiments run during this phase will both prove the quality of the magnet and illustrate baseline capabilities for the NMR user community. As the commissioning phase progresses, more and more magnet time can be devoted to user applications. A strong emphasis will be placed on experiments that require the unique wide bore as well as the 900 MHz field. A discussion of the NMR science priorities for the 900 and the capabilities of a wide bore instrument can be found in the "Attention Users" column by Tim Cross in the Fall 2002 *NHMFL Reports* (**9**(3) 2002).

Having outlined our plans for the commissioning phase above, it is worthwhile to note at this time that any superconducting magnet system is a high tech instrument operated in a very technically challenging environment. In our case, we are dealing with several difficult feats in one combined system: a large bore 900 MHz (equivalent to 21.1 T) superconducting magnet system, a sub-cooled 1.8 K cryostat (only the second such large scale NMR cryogenic system in the world), and finally the exploitation of unique NMR science. During the commissioning phase, we will be operating this system in the permanent cryostat and finding our balance for these combined feats for the first time. We applaud the scientists, engineers, and technicians who have risen to the challenge and are joining their skills towards the common goal of creating the world's first 105 mm bore, 900 MHz NMR system.